Fundamentals and Comparisons for Organic Sample Extract Evaporation

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PREPARE FOR BETTER RESULTS

Abstract

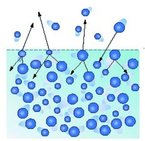
Sample preparation is a key part of the analytical process, contributing to reproducibility and accuracy of the final results. Generally, sample preparation for organic analysis requires the analytes of interest to be first extracted from the matrix. Then cleanup of the extract may be required to remove interferences arising from the matrix. Water is removed during the drying step if it was introduced from the samples. Finally the extract is reduced in volume to accommodate the detection limits needed for the analysis and the ability of the instrument to accommodate a large-volume sample.

The evaporation/concentration step can be achieved with various technologies, including heat, vacuum, and blow-down. We will examine the parameters that go into each of these choices and describe criteria to consider in matching the sample to the technique. Further, solvent recovery has become increasingly important as the number of samples analyzed and the size of individual laboratory locations has increased. The implications for solvent recovery based on the type of evaporation will be discussed.

Introduction

Evaporation

Sample usually consists of organic analytes of varying volatility in a solvent/reagent of known volatility



The key is to efficiently remove the solvent/reagent without losing more than an acceptable amount of the analytes

Evaporation Parameters

- Sparge gas
- Heating the sample
- Vapor removal
- Motion
- Closed system vs. Open system
- Solvent recovery

Do nothing scenario for comparison

- Solvent left in a beaker will evaporate on its own
- Not efficient
- Not controlled
- May occur differently each time depending on ambient conditions

Experimental

Evaporation Parameters:

Sparge Gas

- Gas helps to disrupt the surface equilibrium to promote further solvent molecules escaping into the vapor phase
- Certain action, such as vortex action, is more effective than random turbulence
- The gas should be inert or analyte oxidation may occur
- As concentration progresses it becomes more likely to dislodge analyte molecules, so additional care should be used

Heating

Sample heating to promote evaporation should be

- Quick to heat
- Quick to turn off when heating is done
- Controlled to an optimum temperature for the analyte/solvent combination
- Uniformly distributed around the sample

Heating alternatives

- None (Ambient)
- Hot water/Oil
- Hot plate/Block
- Hot Air
- Steam
- Immersion Heater
- Infrared Light

Vapor Removal

- None
- Fan
- Vacuum
- Vacuum can be used to lower the boiling point of the solvent to facilitate evaporation
- May help to retain low-boiling analytes
- Operator safety is critical!

Motion

- Motion can be used to increase the surface area available for evaporation
- Rotating or centrifuging the sample container are possible ways to do this
- Using sparge gas to create turbulence can also create this effect
- Cooling effect of inert gas limits analyte loss
- Impacted considerably by the shape of the containment vessel (tube)

Open System vs. Closed System

Open System

- Less complicated
- Vapors cannot be efficiently captured
- More versatility in terms of glassware

Closed System

- Less chance of volatile loss, cross contamination
- Vacuum can be used
- Vapors can be recovered
- Less versatility in glassware choice

Solvent Recovery

- In a closed system, solvent recovery is possible because the solvent will provide the bulk of the vapor
- Solvent recovery when large volumes of sparge gas are used or water vapor is co-mingled is not possible
- Environmentally friendly to recover solvent

Results and Discussion

Kuderna Danish

- Used for many years
- Became the early standard for evaporation/concentration
- Uses heat
- Partially closed system
- Reflux action

Other types of systems have been introduced

System	Heat	Sparge Gas	Vapor Removal	Motion	Closed/ Open
Kuderna Danish	Yes	No	None	No	Partially
Rotary Evaporator	Yes	No	Vacuum	Yes	Closed
XcelVap Evaporator	Yes	Yes	Fan	No	Open
DryVap Evaporator	Yes	Yes	Vacuum	No	Closed

Considerations:

- Volume for samples
- Type of samples (more volatile content)
- Laboratory sample load
- Initial investment considerations

DryVap System	XcelVap System
In-line solvent extract drying	Off-line drying required
Handles 15-200 mL solvent extracts	Handles a few mL to 200-mL size extracts
Compatible a variety of Horizon DryVap tubes	Compatible w/most commonly available glassware
Sealed system blanketed with inert gas	Blanketed with inert gas
Can save a number of methods with optional computer and control software	One method saved run to run
Can change methods on the fly for each module	Can change method during a run, but original method is the one saved
Up to (6) samples processed simultaneously	Up to (54) samples processed simultaneously
Evaporation Direct to GC Vials	Accommodates a variety of glass test tubes, KD, and VOA vessels
Vacuum, heat and sparge gas process	Heat and gas process
Direct heat applied (immersion heater)	Heated water bath
Optical endpoint sensor	Timer with adjustable audible alarm
Solvent vapor recovery is possible	All solvent vapors are exhausted no recovery possible
Less than 30 minutes to evaporate 200 mL to 1 mL	50-60 minutes to evaporate 200 mL to 1 mL
More expensive option	Less expensive option
Larger footprint (27.5 x18.75x17.25 inches)	Smaller footprint (12x12.5x22.5 inches)

Conclusions

- Sample preparation is a key step in the analysis process
- Parameters for evaporation and their impact on analysis have been discussed
- Improvements in matching the sample to the evaporation device characteristics can help reduce variability and improve recovery
- Examples for choosing a system based on sample volume, types of analytes, sample load, and initial investment considerations gives guidance on both analytical and business considerations